practical NO. 1

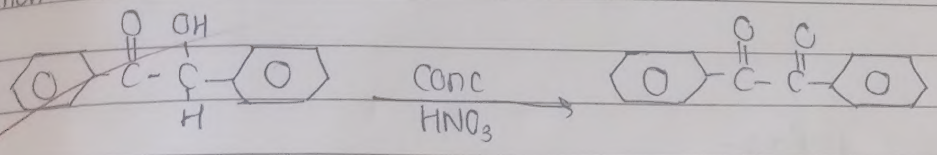
Benzoin ⟶ Benzil ⟶ Benzilic acid.

part-I

Aim:- preparation of Benzil from Benzoin.

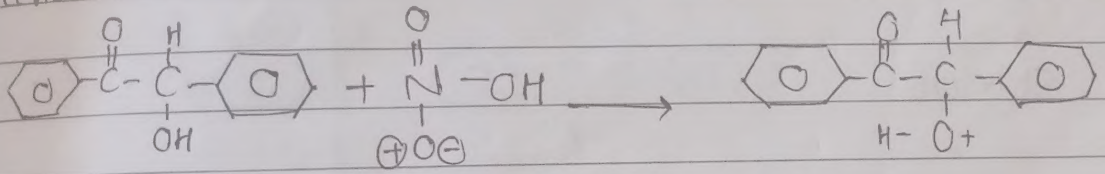
Requirement:- Conical flask, burner, Condensers, Benzoic, con HNO_3 etc.

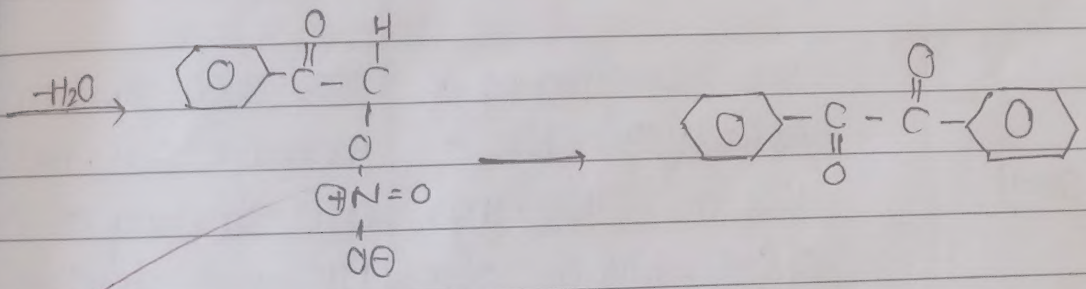
Reaction:



Benzoin Benzil

Mechanisum

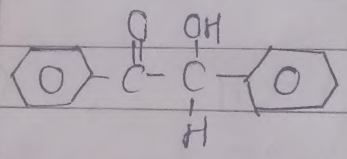
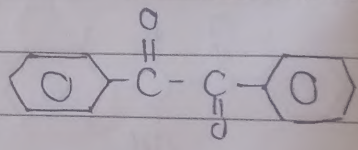




Stoichiometric Calculation.

	Benzoin	Con. HNO_3
Molecular formula	$C_{14}H_{12}O_2$	HNO_3
Molar weight	212	63.
Molar ratio	1	1
Volume	-	0.75
No. of moles	0.018	0.018
Q (gm)	4 gm	1.34gm

Spectral Data :-

	Benzoin	Benzil
Structure		
IR (cm-1)	3100-3000 streching of Ar(-H(sp^2) - 1660 - 1550 cm^{-1} streaching of C=C . 1715 cm^{-1} stiec-hing of Ketonic C=O	3100-3000 streching o Aromatic C-H . 1660 - 15 cm^{-1} streching of C=O 1715 cm^{-1} streching of Ketonic C=O .
^1H NMR	Benzene ring 6.5-8.5 8H R-OH : 1-6.	Benzene ring 6.5-8.5 m 8H.

procedures:-
Take 4 gm of benzoin and 20 cm^3 of con HNO_3 in Conical flask.
Heat it on boiling water bath with occasional stirring until the evolution of oxides of nitrogen has stopped.
pour the reaction mixture in 20 cm^3 of cold water and stir well.
The oil crystallises and yellow solid is obtained.
Filter the Crude product at the suction pump wash with cold water to remove HNO_3 and dry Note the yield of the crude benzil purify the portion of the product and take it MP.

Calculation:

Weight of Crude product: 3.7 gm
Melting point : 94°C

Theoretical yield:-
212 gm of benzoin = 210 gm of Benzil.
4 gm of benzoin = x gm of Benzil

$$x = \frac{210 \times 4}{212}$$

$$\boxed{x = 3.96 \text{ gm}}$$

percentage yield: $\frac{\text{practical yield}}{\text{Theoretical yield}} \times 100$

$$= \frac{3.7}{3.96} \times 100$$

$$= 96\%$$

tion of benzil from benzoin

Date: 26/04/24

Rf value = Distance travelled by solute / Distance travelled by solvent

= 3.5 / 4

= 0.87

MSDS Data:-

Name of compound	Health Hazards	First Aid.
Benzoin	many causes eyes and skin irritation. Causes respiratory tract irritation Harmful of swallowed.	Flush the eyes and skin with plenty of water.
Nitric acid	Causes seven eyes and skin burons. may causes respiratory tract and digestive tract irritation Very harmful if swallowed.	Rinse the eyes with plenty of water flush the affected area of skin with plenty of water.
Ethyl alcohol	Cause eye skin respiratory tract irritation.	Flush eyes and skin with plenty of water.

Result:-

Theoretical

Melting point

Weight of

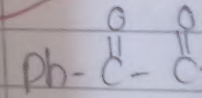
percentage

Aim:- prep

Requirement

Reaction.

Mechanism



Result:-

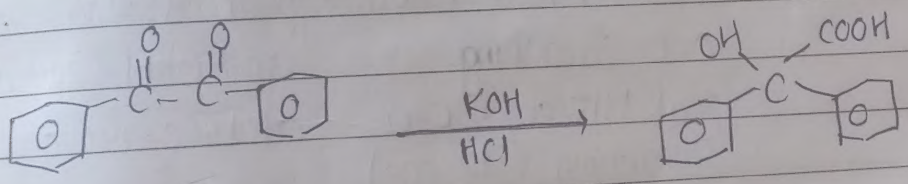
Theoretical yield: 3.96 gm
Melting point : 94
Weight of the product: 3.7
Percentage yield: 96%.

Part - II

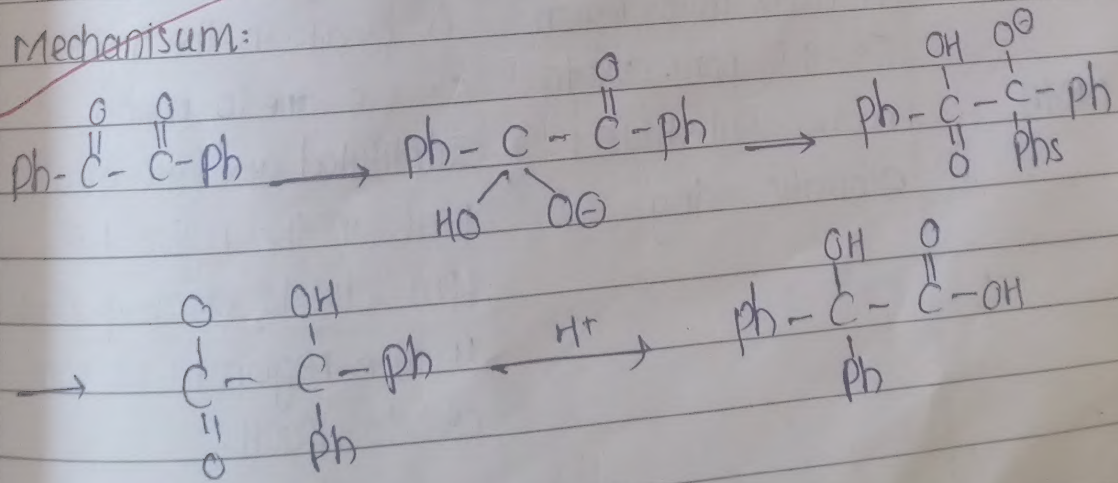
Aim:- preparation of Benzilic acid from benzile.

Requirement: Conical flask, 1g Benzile, 150 cm^3 Water bath cold water Filter paper.

Reaction.



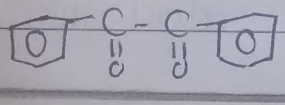
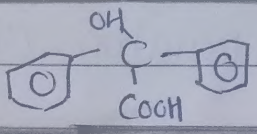
Mechanisum:



Stoichiometric Calculation:

	Benzil	KOH
Molecular formula.	$C_{14}H_{10}O_2$	$K_1O_1H_1$
Molecular Weight	210	56
Molar ratio	1	1
φ (gm)	1	0.280
Volume	-	-
N.O. of moles.	0.005	0.005

Spectral Data:-

	Structure	
		
IR (cm^{-1})	peak at 3100-3000 cm^{-1} C-H (SP^2) Streaching peak at 1600-1550 cm^{-1} C=C streaching. peak 1715 cm^{-1} C=O Streaching (Ketone)	peak at 3600-3200 cm^{-1} O-H Streaching peak at 3100-3000 cm^{-1} C-H (SP^2) Peak at 1660-1550 cm^{-1} C=C.
^1H NMR	A peak in the region 6.5-8.5 ppm. due to mono substituted aromatic ring	A peak in the region 6.5-8.5 due to mono substituted aromatic ring peak in the region 1.6 ppm due to OH group peak in the region 10-12 ppm due to COOH.

procedure:-
Reflux the mixture 1 gm of benzil 1 g of KOH 5 cm^3 of water and 2.5 cm^3 of alcohol on heat for 10-15 mint
pour the contain of the flask into a pore dish allow it to cool
The potassium salt of benzilic acid and crystallized.
Filter off the crystal at the pump and wash with ice cold alcohol.
Dissolved the potassium salt in about 10 cm^3 of water and acidify with con. HCl to get present Wash the precipitate and recrystalline with hot water
take the melting point of product.
check the purity by TLC. Submit the product.
Calculation.

Theoretical yield:-
210 g of benzil : 228gm of benzilic acid.
1 g of benzil : x gm of benzilic acid

$$x = \frac{228 \times 1}{210}$$

$x = 1.08$ Benzilic acid.

percentage yield:-

$$\frac{\text{practical yield}}{\text{Theoretical yield}} \times 100$$

$$\frac{0.6}{1.08} \times 100$$

$= 55\%$

Practical - 1 Preparation

Preparation of benzil from ben...

Rf value:-

$$\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$$

$$= \frac{3.2}{5}$$

$$= 0.64$$

non-polar (n-hexane) - 80%
polar (ethyl acetate) - 20%

Result:
Theoretical yield : 1.08 gm
Percentage yield : 5%
Weight of the product : 0.6gm
Rf value 0.64
Solvent System : n-hexane - 80%
ethyl acetate - 20%

MSDS Data:-

Name of Compound	Health Hazards	First Aid.
Benzil	eyes and skin irritation Causes respiratory tract irritation Harmful is swallowed	Flush the eyes are skin skin with waters. Call poison center immidiately
potassium Hydroxide.	Causes severe skin burns and eyes damage Causes respiratory and digestive tract irritation.	Flush the eyes with plenty of water and move to air atleast 15 mint
HCl	Causes severe skin burns and eyes damage Causes respiratory tract irritation.	Flush the eye plenty of waters and move to attleas 15 mint.

25/4/23

Practicle No. 2

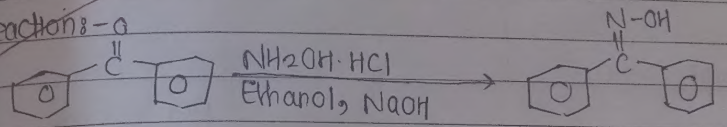
Benzophenone ⟶ Benzophenone oxime ⟶ Benzanilide.

part-I

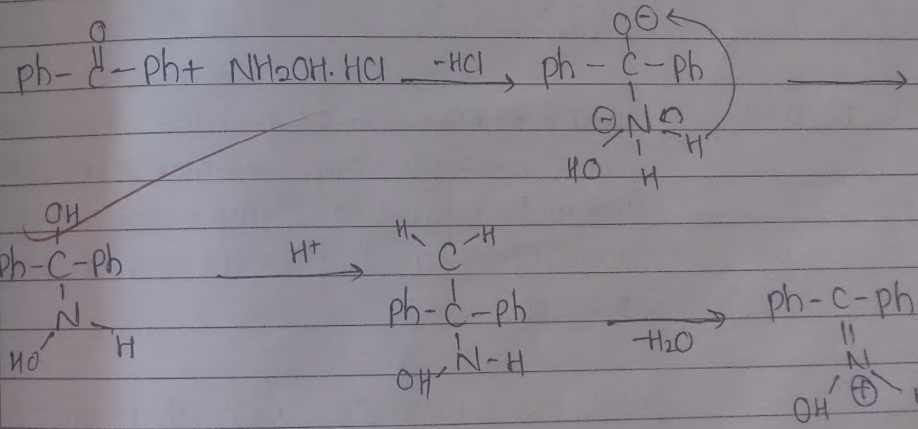
Aim:- Preparation of Benzophenone oxime from Benzophenone.

Requirment:- RBF, burners, water bath, benzophenone, rectified spirit, hydroxylamine hydrochloride.

Reaction:-



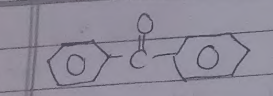
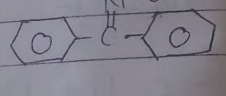
Mechanisum:-



Stochiometric Calculation.

	Benzophenone	Hydroxyl amine Chlo
Molecular formula	$C_3H_{10}O$	NH_2OCl
Molecular weight	182	69.5
Molar Ratio	1	1
Quantity (gm)	3	1.112
Quantity (cm^3)	-	0.6825
no. of moles	0.016	0.016

Spectral Data:

	Benzophenone	Benzophenone oxime
Structure		
IR (cm^{-1})	peak in the region 3100-3000 cm^{-1} C-H sp^2 Streaching peak in the region 1800-1600 cm^{-1} C=O Streaching of Ketone.	peak in the region 3100 3000 cm^{-1}(sp^2) Broad peak region 3600-3200 cm^{-1} O-H Streaching
	peak in the region 6.5-8.5 ppm SCH)-m-monosubsubsi tuted Aromatic ring peak in the region 6.5-8.5 ppm SH(m) monosubsituted Aromatic ring	peak in the region 6.5-8 ppm 5(H)m monosubstitu Ar. ring peak in the reigion 6.5-8.5ppm. 5H(m) mono-Substituted ring

procedure:-
Take 3g of Benzophenone 2 grams of hydroxylamine hydrochloride and 6 cm^3 of restified spirit.
Add 1 cm^3 of water to this mixture add 2.5 grams of solid NaOH in portion of 0.5 grams with constant shaking
the reflux the reaction mixture 10 mint. cool and pours the constant of the flask into a mixture of pours the content of the flask into a mixture of 10 cm^3 of conc. HCl and add 60 cm^3 of water.
Filter the preapitated and dry the product.
Note the yield of crude benzophenone oxime purify the portion of the product and take it melting point.

Observation:

Weight of crude product: 2.44 cm^3
Melting point: : 142°C

Calculation:-
Theoretical yield:-
182g of Benzophenone = 197g of Benzophenone oxime
3g of Benzophenone = x

$$x = \frac{197 \times 3}{182}$$

$$x = 3.2g.$$

Percentage yield = $\frac{\text{Observed yield}}{\text{Theoretical yield}} \times 100$

$$\frac{0.44}{3.2} \times 100 = 75\%$$

Result :-

weight of crude product :- 0.114 gm
percentage yield :- 75%
Theoretical yield :- 3.29
Melting point :- 142° C.

MSDS Datas :-

Name of compound	Health Hazard's	First Aid.
Benzophenone.	Causes eyes and skin irritation Causes gastrointestinal irritation. Causes respiratory tract irritation.	Flush eyes with plenty water for at least 15 mint move in fresh air
Ethanol	Causes eye and skin irritation causes respiratory	Flush eyes with plenty waters move to fresh air
Hydroxyl amine hydroxy chloride	causes eyes and skin irritation Harmful if swallowed many cause respiratory irritation	Flush eyes with waters and move to fresh air at least 15 mints.

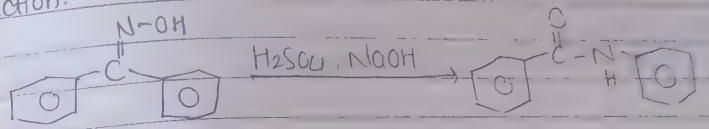
08

Part-II

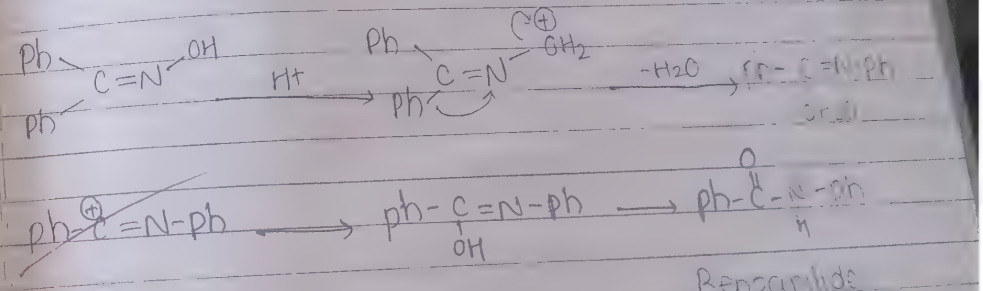
Aim :- preparation of Benzanilide from Benzophenone oxime

Requirements :- Beakers, Phenzophenone oxime, Water, salt, diethyl ether, penta Chloride

Reaction :-



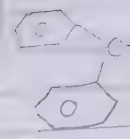
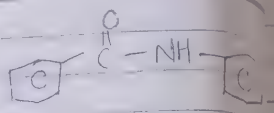
Mechanisum :-



Stochometric Calculation :-

	Benzophenone oxime	Conc H_2SO_4
Molecular formula	$C_{13}H_{11}NO$	H_2SO_4
Molecular weight	197	98
Molar ratio	1	1
Q (gm)	1	0.49
Volume	-	0.26
no. of moles.	0.005	0.005

FOR E

	Benzophenone oxime	Benzanilide
Structure:-		
IR (cm⁻¹)	peak in the region 3100-3000 cm⁻¹ C-H (sp^2) Streching Broad peak in the region 3600-3200 cm⁻¹	peak in the region 3100-3000 cm⁻¹ C-H (sp^2) Streaching Sharp peak in the region 3600-3200 cm⁻¹
	peak in the region 1680-1500 cm⁻¹ C=C Streching	peak in the region 1700-1660 cm⁻¹ C=O streaching
¹H NMR	peak in the region 6.5-8.5 ppm. peak in the region 1-6 ppm. H(s) OH proton.	peak in the region 6.5-8.5 ppm 5H (m) monosubstituted Ar-ring monosubstituted Aromatic ring peak in the region ppm Ar-NH proton 1H(s)

Procedure:-

Take 1 gram of Benzophenone oxime add the cool solution of conc. H_2SO_4 in Water (1.6 cm^3 Conc. H_2SO_4 to 2.0 cm^3 water) Shake the flask and warm it gently to begin the reaction. Remove the flask from burners and allowed reaction. Once the reaction subsides. Cool the content flask and then add 4 cm^3 of water.

Add 10N NaOH solution to reaction mixture dropwise maintaining Compound by using 3 portion of carbon tetrachloride. yellow oil is obtained which solidified on cooling purify the compound by recrystalisation

Note the yield of Benzanilide. take the melting point of the the product.

Check the purify the product by TLC Submit the dried product.

Observation:

Weight of the Benzanilide :- 0.2 gm

Melting point :- 162°C

Calculation:

Theoretical yield :-

197 g of Benzophenone = 197g of Benzanilide oxime.

1g of Benzophenone oxime : x g of Benzanilide

$$x = \frac{197 \times 1}{197}$$

x = 1g of benzanilide.

Percentage yield: $\frac{\text{weight of the product}}{\text{Theoretical yield}} \times 100$

$= \frac{3.2}{1} \times 100$

$= 20\%$

Rf value:- $\frac{\text{Distance traveled by solute}}{\text{Distance traveled by solvent}}$

$\frac{3.2}{40}$

$= 0.8$

Result:-

Weight of the crude product	:- 0.2 grams
Theoretical yield	:- 1g
percentage yield	:- 20%
Melting point	:- 162°C
Rf value	:- 0.8
Solvent System	:- 60% (non-polar)
	40% (polar)

MSDS Data:

Name of compound	Heath Hazard	First Aid
Benzophenone oxime	may cause eyes irritation. may cause digestive tract irritation. Harmful if swallowed	Flush eyes with plenty of water get medical aid
Diethyl ether	may cause skin dryness. Harmful if swallowed	Flush eyes and skin with plenty to water get medical aid
phosphorous penta Chloride	Chemical burns of the respiratory tract Harmful if swallowed	get medical aid immediately if breating is difficult give oxygen.

Rf =

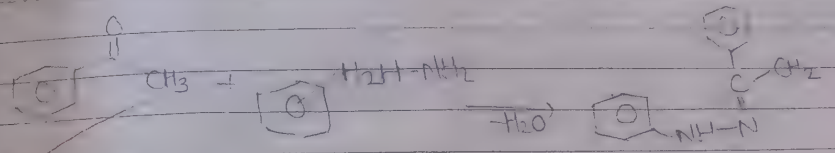
2.4 / 2

Part-I

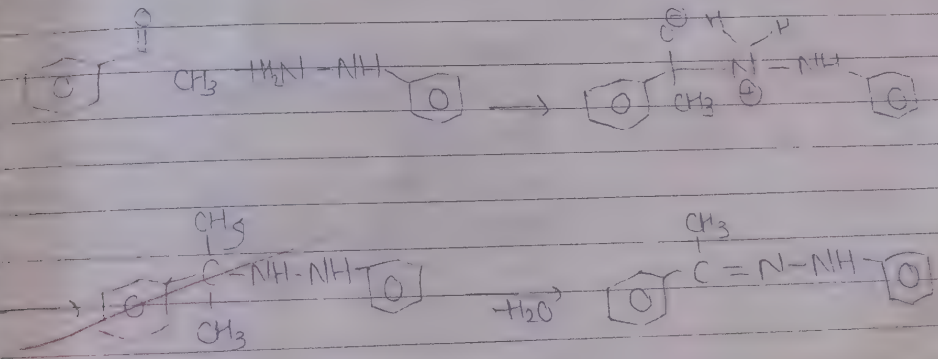
Aim:- Preparation of Acetophenone phenylhydrazone From Acetophenone.

Requirement: Beakers, Acetophenone phenylhydrazine glacial acetic acid etc.

Reaction:



Mechanism:



Procedure:-

Take 5 cm^3 acetophenone and 5 cm^3 phenyl hydrazine with 30 cm^3 of ethanol four drops of glacial acetic acid is formed

Warm the reaction mixture till the solid product is formed cool and filter and wash the product with dilute followed by about 10 cm^3 of cold distilled spirit Dry the product

Note the yield of crude acetophenone phenyl hydeazone purify recrystallization of the product and take it's M.P

Observation:-

product: 6.9 gm

: 186°C

Calculation:

120 gm of acetophenone 210 gms of acetophenone phenyl hydrazone

5.14 gm of acetophenone : x gm of acetophenone phenyl hydrazone

$$x = \frac{210 \times 5.14}{120}$$

Percentage yield: $\frac{\text{Observed yield}}{\text{Theoritical yield}} \times 100$

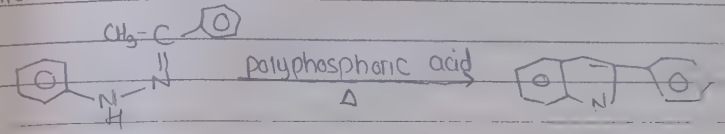
Name of compound	Hazards	First Aid
Acetophenone	Causes eye and skin irritation Causes respiratory tract irritation	Flush the eye and skin with plenty of water move to fresh air and get medical Aid
Phenyl hydrazine	Causes eye and skin irritation Causes respiratory tract and digestive tract irritation	Wash the eyes clean with plenty of water for 15 min Wash skin and eyes with plenty of water
Ethyl Alcohol	Causes eye and skin irritation Causes respiratory tract irritation	Wash skin and eyes with plenty of water
Acetic acid	Causes severe skin burns and eye damage may be harmful	With affected area with plenty of water move to fresh air

Part - II

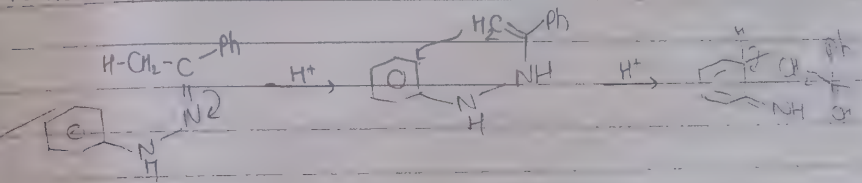
Aim: preparation of phenyl indole from acetophenone phenyl hydrazone.

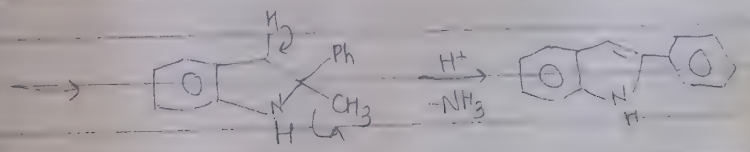
Requirement: Acetophenone phenyl hydrazoic, Beaker, cold water, water bath.

Reaction:



Mechanism:





2-phenyl indole

Stoichiometric calculation

	$C_{14}H_{14}N_2$	Hn + 2PnO3nen
Molecular formula	$C_{14}H_{14}N_2$	Hn+2PnO3nen
Molecular weight	210	337.93
Molar Ratio	1	1
Q (gm)	1	1.588
Volume	–	0.756
No of moles	0.0047	0.0047

Spectral Data:

	Acetophenone phenyl Hydrazone	2-phenyl Indole
Structure		
IR (m^{-1})	peak in the region 3100-3000 cm^{-1} C-H (SP^2) Streaching Sharp peak in the region 3600-3200 cm^{-1}	Peak in the region 3100-3000 (SP^2) Stretching peak in the region 3600-3200 cm^{-1}
^1HNMR	peak in the region 6.5 to 8.5 ppm peak in the region 8.5 ppm	peak in the region 6.5 to 8.5 (5H) m (monosubstituted) peak 6.5 to 8.5 5H

Take 1 gm of crude acetophenone
3.5 cm^3 of polyphosphoric acid
Heat the mixture with constant stirring and maintain temperature 100-120 for 4min
Filter the product and stir well to get product purity the small portion and take MP

Weight of the Product : 0.4 gm
Melting point : 190°C

210 gm of acetophenone phenylhydrazone : 193 gm of 2-phenyl indole
1 gm of acetophenone phenylhydrazone : x gm of 2phenyl indole

$$x = \frac{193 \times 1}{210}$$

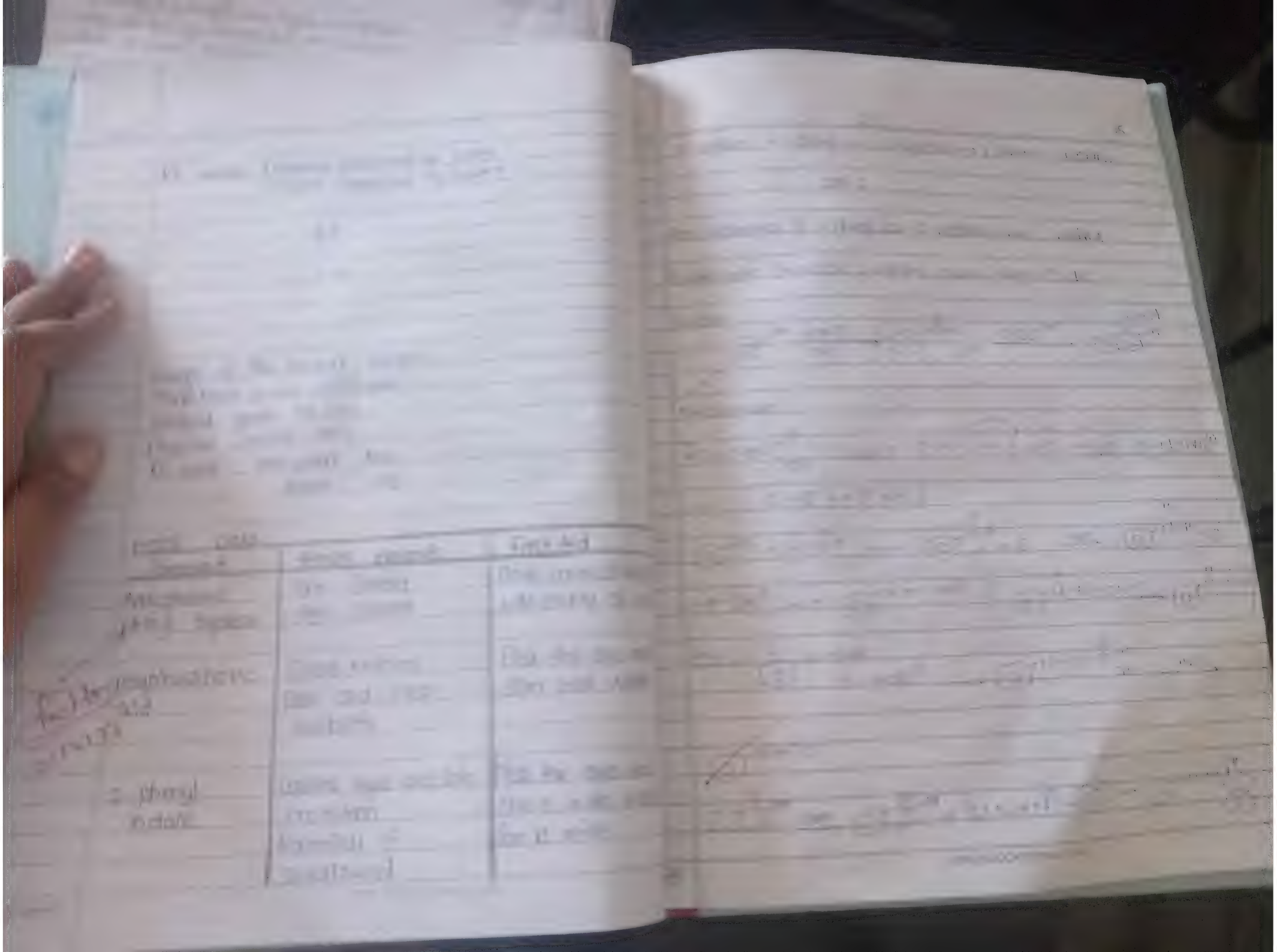
$$x = 0.919$$

$$= \frac{\text{Weight of the product}}{\text{theoretical yield}} \times 100$$

$$= \frac{0.4}{0.919} \times 100$$

$$= 43.53\ \%$$

FOR EDUCATIONAL



Rf value = Distance travelled by solute / Distance travelled by solvent

3.4 / 4.8

0.74

Weight of the product: 0.4gm
Theoretical yield = 0.919 gms
practical yield: 43.53%
physical Constant: 190°C
Rf value: non-polar 80%
polar 20%

MSDS Data:

Compound.	Health Hazards	First Aid
Acetophenone phenyl hydraze	skin Contact eye contact	Flush immediately with plenty of
polyphosphoric acid	Causes redness pain and poor visibility	Flush the eye or skin with
2-phenyl indole.	Causes eyes and skin irritation Harmfull if swallowed	Flush the eyes and skin in with for 15 mint

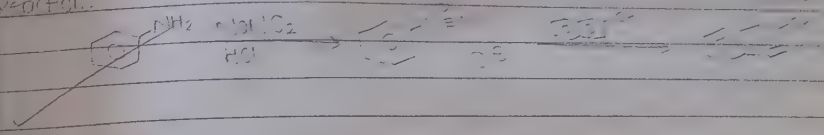
2-Naphthol → 1-Phenyl azo-2-naphthol → 1-amino-2-naphthol

part-I

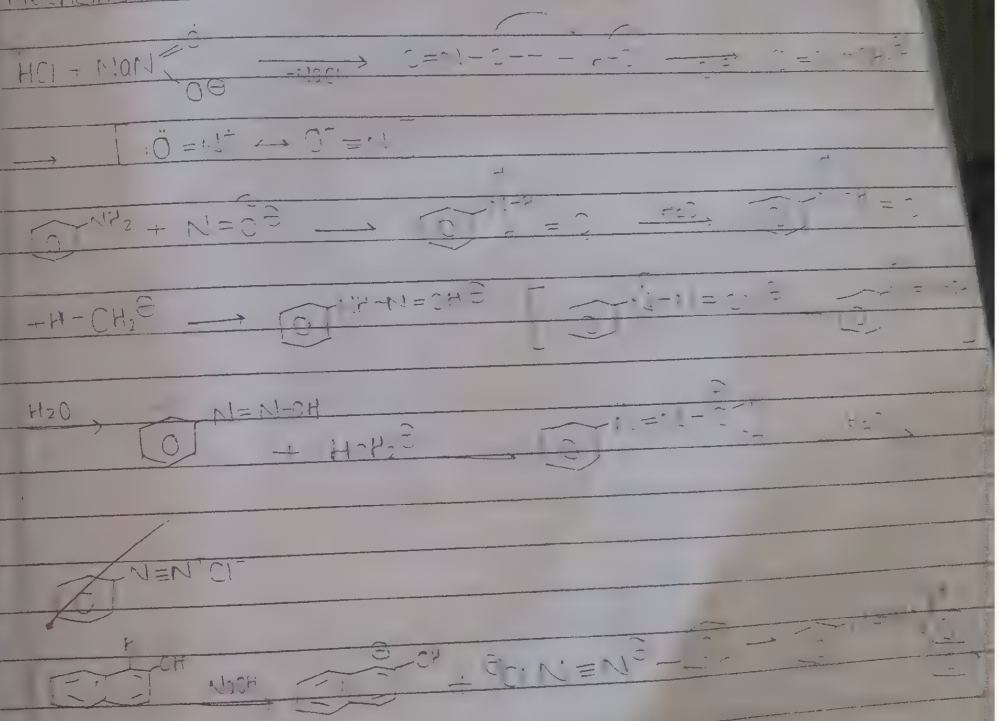
Aim: preparation of 1-phenyl azo-2-naphthol from 2-nitro

Requirements: Conical flask, 2-naphthol, aniline, NaOH sol' etc

Reactor.

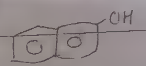
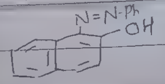


Mechanism:



	2-Naphthol	Aniline (1.0297 g/cm³)
[illegible] formula	$C_{10}H_8O$	C_6H_7N
[illegible] weight	144	93
[illegible]	1	1
[illegible] in gm	5	0.041 x 93 = 3.81
[illegible] in cm³	–	3.81 – 1.0297 = 3.7
No. of Moles	0.041	0.041

[illegible] Data:

	2-napthol	1-phenyl-azo-2-naphth[illegible]
Structure		
IR [illegible]	① peak in the region 3100-3000 cm⁻¹ C-H sp² stretching ② Broad peak 3600 cm⁻¹ 3200 cm⁻¹ OH stretching ③ 1650-1500 cm⁻¹ stretching C=C atom. ④ 1300-1050 cm⁻¹ stretching C-O group.	① 3600-3200 cm⁻¹ stretch of O-H group. ② 1666-1500 cm⁻¹ stretch of C=C. ③ peak in the region 3100-3000 cm⁻¹ C-H (SP²)
¹H NMR	Aromatic Aro-H 6.5-8.5 ppm	Aromatic Aro-H. 6.5-8.5 p[illegible]

Procedure: Dissolve [illegible] of aniline [illegible] the mixture by addition of [illegible] of [illegible] [illegible] in [illegible] of water, [illegible] mixture below [illegible].

prepare a solution of [illegible] soln and cool this solution [illegible] and to it, add cold diazonium salt [illegible] the mixture to stand [illegible] stirring. filter the product [illegible] with water and dry. Note the yield of [illegible] purify small portion and take melting point.

Observation:

Weight of Crude product [illegible]

Melting point of the product. [illegible]

Calculation:

Therotical yield:

93 g of aniline = 248 g of 1 phenyl azo-2-naphthol

3.81 g of aniline = x g of 1 phenyl azo-2-naphthol

$$93 \times x = 248 \times 3.81$$

$$x = \frac{248 \times 3.81}{93}$$

$$x = 10.16\,g.$$

yield x100

$\frac{8.00}{10.16}$ x100

78.74%

(practical yield) : 8.00 g.

[illegible] yield : 10.16 g

[illegible] yield : 78.74%

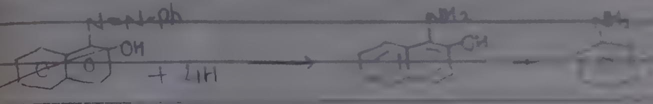
[illegible] point : 135°C

Health Hazards	First acid
[illegible] causes skin irrita- tion. Systemic effects occur from all routes of exposure and can include.	Quickly remove contaminated clothing. Wash contaminated skin with large amounts of soap and water.
Causes eye, skin irritation may cause irritation of the digestive tract. respiratory tract irritation.	Immediately flush with plenty of water for at least 15 min. Get medical aid immediately.

Reaction

Mechanisum.

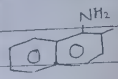
$SnCl_2 + 2HCl \longrightarrow SnCl_4 + 2H +$ [illegible]



Stoichiometric Calculation

	[illegible]	[illegible]
Molecular formula	[illegible]	[illegible]
Molecular weight	[illegible]	[illegible]
Molar ratio		
Quantity in gm		[illegible]
No. of Moles	[illegible]	[illegible]

Practical Data

	1-Phenyl azo-2-naphthol	1-amino-2-naphthol
Structure		
IR cm^{-1}	①peak at region 3100-3000 cm^{-1} C-H sp^2 stretching ②Broad peak 3600-3200 OH stretching. ③peak in the region 1680-1500 cm^{-1} C=C Stretching ④peak ~1200 cm^{-1} C-N Stretching.	①peak in the region 3100-3000 CH sp^2 stretch ②Broad peak 3600 cm N-H stretching. ③peak in the region 162 1500 cm^{-1} C=C Stretchin ④peak in the region 1300-1050 cm^{-1} C=O Stretch
	6.5-8.5 ppM Ar-ring present. 4-8 ppM Ar-OH proton.	6.5-8.5 ppM Aroma ring present.

Procedure Reflux the mixture of 1gm of 1-phenyl azo-2-
and 10 cm^3 of rectified Spirit till the azo compound is di
Separately prepare a reducing mixture of 6.7g tin (II)
in 20 cm^3 Conc HCl add this reducing mixture to 1-pheny
2-naphthol and reflux for fullther 30 mint. the solution
a brown colour. Decant the solution into a beaker and
ice bath till 1-amino-2-naphthol hydrochloride Separate
greyish white white crystals. filter the product Using Suc

Wash with dil HCl and dry. Note the yeild of 1-amino-2-naphthol and take the MP of product and cheak TLC.

Observation:

Weight of Crude product: 0.4g

Melting point of the product: 250°C

Calculation:

Therotical yield:

248 g of 1-phenyl azo-2-naphthol = 159 g of 1-amino-2-naphthol

1g of 1-phenyl azo-2-naphthol = x g of 1-amino 2-naphthol

$$248 \times x = 159 \times 1$$

$$x = \frac{159 \times 1}{248}$$

$$x = 0.64\ g.$$

percentage yield:

$$= \frac{\text{observed yield}}{\text{Therotical yield}} \times 100$$

$$= \frac{0.4}{0.6} \times 100$$

$$= 62.5\%$$

Rf Value: $\frac{\text{Distance traveled by Solute}}{\text{Distance traveled by Solvent}}$

$$= \frac{3.2}{3.5} = 0.9$$

IF conical

Result:
Weight of the product (observed yield) = 0.4g
Theoretical yield = 0.6g
percentage yield = 62.5%
physical constant (M.P) = 250°C
RF value = 0.9
(solvent system = 90% n-hexane and 10% ethyl acetate)

MSDS Data:

Name of Compound	Health Hazards	First acid.
① Tin (II) chloride	① Cause eye burns ② Cause skin burns ③ Harmfull if swallowed	Flush the eye and sk... with plenty of wate... Get medical acid imm... diately.
HCl	very hazardous in Cause of ingestion	flush the eye and s... with cold water.
Alcohol	Causes eyes, skin and respiratory tract imitation	flush eye and skin with plenty of wate...
free $C_6H_5N_2$ 1-phenyl-aza-2-naphthol.	may cause an allergic skin reaction Suspected of causing Cancer	flush the skin with plenty of water get medical immediately

(6)

Anthracene → Anthraquinone → Antrone.

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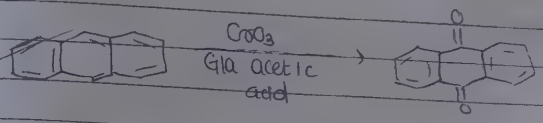
Part-I

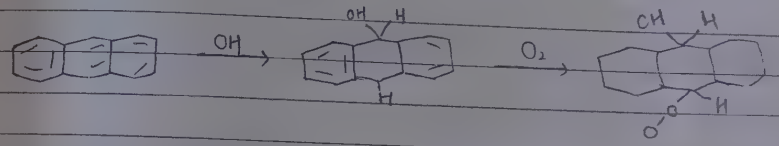
Aim: Preparation of anthraquinone from Anthracene

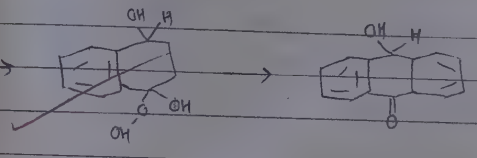
Requirement: Beaker glacial acetic acid, CrO_3 Anthracene. Cold water

Reaction:

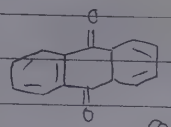


Mechanism:





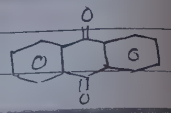
semiquinone



① Dinone (9,10) anthra

Stoichiometric Calculation:-

	Anthracene	CrO_3
	~~$C_{14}H_{10}O$~~ $C_{14}H_{10}$	CrO_3
Molecular Formula	178	99.99
Molecular Weight	1	1
Molar Ratio	55	2.89
ρ (gm)	-	-
ρ (cm^3)	0.028	0.028
no of moles		

	Anthracene	Anthraquinone
Structure		
IR (cm^{-1})	Region 3100-3000 C-H (SP^2) Streching Region 1680-1500 cm^{-1} C=C Streching	Region 3100-3000 cm^{-1} C-H (SP^2) Stre Region 1680-1500 cm^{-1} C=C Streachi
^1H NMR	Aromatic C-H at region 6.5-8.5 ppm	Aromatic C-H at region 6.5-8.5 pp

procedure:-

Dissolve 5 grams of anthracene by refluxing it with 75 cm^3 of glacial acetic acid.

Prepare solution of 3.2 gms of CrO_3 in 25 cm^3 of glacial acetic acid.

Add the oxidising agent to the anthracene solr and then reflux for further 8-10 minutes.

When all anthracence get recicted completely cool the reaction.

Mixture and pour into 250 cm^3 of cold water with stirring. The product is seprate by Filteration. Wash with hot water and dry

Note the yield of Crude Anthraquinone purify the small portion of the product and take it MP.

observation:-

Weight of the product: 4.7 gms

Melting point : 283°C

Calculations:-

Theoretical yield:-

178 gm of Anthracene = 208 gm of Anthraquinone.

3 gm of Anthracene x gm of Anthraquinone

$$x = \frac{208 \times 5}{178}$$

$$x = 5.84 \text{ gm}.$$

Percentage yield:- $\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$

$= \frac{4.7}{5.84} \times 100$

$= 80\%$

Result:-
Theoretical yield :- 5.84gm
Weight of product :- 4.7
Percentage yield :- 80%
physical constant: 283°C.

Name of Compound.	Health Hazards.	First Aid.
Anthracece	eye contact skin contact Inhalation.	Rines immediately with plenty of Rins with plenty of water at least 15 minutes get attention move to the fresh air
Glacial acetic acid	eye contact skin contact Inhalation	Rinse cauthously with Waters for Several mint. Remove lenses take of immediately all contaminated clothing Rinse skin with water.

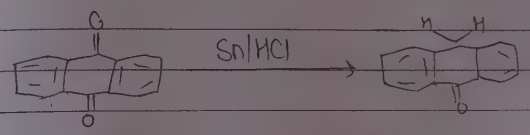
Anthraquinone	Causes eyes and skin irritation. may also causes resp iratory irritation Inhalation	Immediately flush eyes with plenty of water get medical attention Immediately flush eyes with plenty of water move to fresh air if not breathing give artificial respiration.

part-II

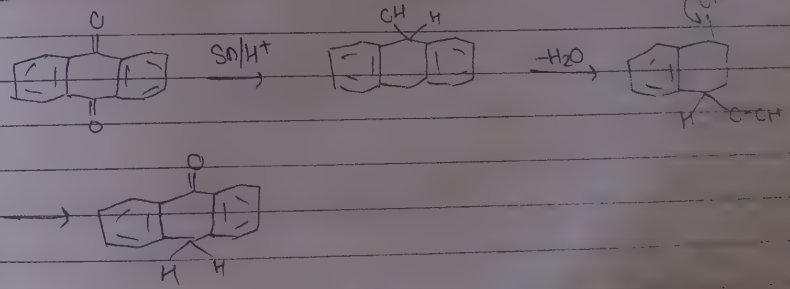
Aim Preparation of Anthrone from Anthraquinone.

Requirements: Beakers, glacial acetic acid, Anthraquinone, con HCl

Reaction:



Mechanism:



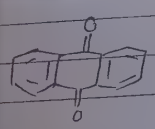
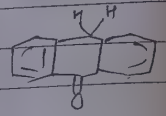
...preparation of benzil from benzoin 26/04/24

... conc HNO_3 in conical
... H_2O bath with stirring until ...

Stoichiometric calculation:

	Anthraquinone	Tin
Molecular formula	$C_{14}H_8O_2$	Sn
Molecular weight	208	188
Molar ratio	1	1
(gm)	1 gms	0.56
No. of moles	0.0048	0.0048.

Spectral Data:

	Anthraquinone	Anthrone.
Structure		
IR cm^{-1}	peak region 1680-1500 cm^{-1} C=C streaching peak region 1800-1660 cm^{-1} C=O streaching peak region 3100-3000 cm^{-1} (CH) sp^2 streaching	peak region 1680 1500 cm^{-1} C=C streaching peak region 1800-1660 cm^{-1} C=O peak region 3100-3000 cm^{-1} (C-H) sp^2
H' NMR	peak region 6.5-8.5 ppm	peak region 6.5-8.5 ppm.

Procedure:-

Reflux the reaction mixture of 1 gm anthraquinone 5 grams of granulated tin and 35 cm^3 of glacial acetic acid for 15-30 mint

cool and then add dropwise 13 cm^3 of Con HCl to it.

If all anthraquinone does not dissolve than add some more granulated tin and HCl

Filter and wash with water and dry by pressing between the filter papers.

Recrystallise it from a mixture of benzene and petroleum ether

Note the yield of pure Anthrone. take the TLC and donde product.

observation:-

Weight of product:- 0.2gms

physical constant :- 155°C.

Calculation

Theoretical yield:-

208 gms of Anthraquinone = 149 gms of Anthrone

1 gms of Anthraquinone = x gms of Anthrone

$$x = \frac{194 \times 1}{208}$$

$$x = 0.933 \text{ gm.}$$

Percentage yield :- $\frac{\text{Weight of product}}{\text{Theoritical yield}} \times 100$

$= \frac{0.2}{0.933} \times 100$

$= 21.4\%$

RF value: $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$= \frac{5.3}{6.2}$

$= 0.85.$

Result:

Weight of the product :- 0.2 gms
Theoritical yield :- 0.933 gms
percentage yield :- 21.4%
RF valve :- 0.85
physical Constant :- 155°C
Solvent System :- n-hexane = 60
ethyl acetate = 40.

MSDS Data:-

Name of Compound.	Health Hazards	First Aid
Anthraquinone.	Skin Contact eyes Contact Inhalation	Immediately flush skin with plenty of water immediately flush with eyes for at least 15 mint
Glacial acetic acid.	Skin Contact eyes Contact Inhalation.	Rines with water for several mint remove Contact inses. Immediately flush eyes.

26/4/23

Practical No. 6.

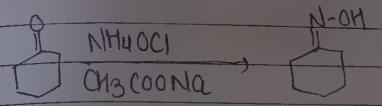
Part - I

Cyclohexanone ⟶ cyclohexanone oxime ⟶ Caprolactam.

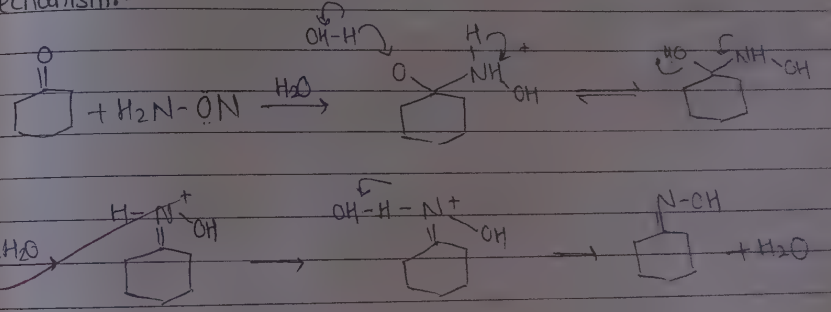
Aim:- preparation of cyclohexanone oxime from cyclohexane.

Requirements- Beaker, ice bath, hydroxylamine, hydrochloride sodium acetate, distilled water cyclohex

Reaction:-



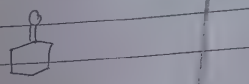
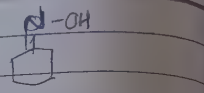
Mechanism:-



Stoichiometric Calculation:-

	Cyclohexanone	NH_4OCl
Molecular formula	$C_6H_{10}O$	NH_4OCl
Molar ration	1	1
q_1 (gm)	4.7 gms	3.312
q_1 (cm^3)	5 cm^3	-
no of moles	0.048	0.048

Spectral Data:

	cyclohexanone	cyclohexanone oxime
Structure		
IR cm^{-1}	C-H (sp^2) Streaching at 3100-3000 cm^{-1} C-H (sp^3) Streaching at 3000-2900 cm^{-1} C=O Streaching at 1800-1660 cm^{-1}	C-H (sp^2) Streaching 3100-3000 cm^{-1} C-H (sp^3) Streaching 300-2000. C-H Streaching 3600 3200 cm^{-1}
H'NMR	Ar-H at region 6.5-8.5 ppm	Ar-H at region at 6.5-8.5 ppm

procedure:-
Dissolve mixture of 3.5 gms of hydroxylamine hydrochloride and 4.2 gms of sodium acetate in 20 cm^3 of water in a conical flask.
add 5 cm^3 of cyclohexanone in small portion.
shake the flask well and cool it in cold water.
Crystal of cyclohexanone in are formed.
Collect the crystal by filtering cold solution using Buchner Funnel.
Wash the crystal with small portion of cold water than dry in air
Note the yield of crude cyclohexanone oxime and purify the small portion of product and take melting point

Calculation:-
Theoretical yield:-
98 gms of cyclohexanone = 113 gm of cyclchexanone oxime
4.74 gms of cyclohexanone = x gm of cyclohexanone oxime

$$x = \frac{113 \times 4.74}{98}$$

$$x = 4.7 \text{ gms.}$$

Percentage yield:- $\frac{\text{yield of the product}}{\text{Theoretical yield}} \times 100$

$$= \frac{3.2}{5.47} \times 100$$

$$= 64\%$$

yields - 5.17 gms
... moisture - 2.2 gms
... yield = 64%
... point = 88°C

MSDS Data:-

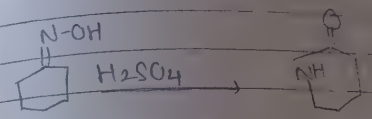
Compound	Health Hazards	First Aid
cyclohexanone	eyes contact skin contact inhalation	Rinse immediate with plenty of water Wash of immediately with plenty of water move to the fresh air and get medication.
Sodium acetate	Skin Contact eye Contact swallowing	Wash affected area with soap and water protect unexposed eyes. Remove lenses. Rinse mouth throughly discomfort or vomiting persists.
Hydroxylamine hydrochloride	Skin contact eye contact Ingestion	Wash off immediately with plenty of water Rinse immediately with plenty of water Do not induce vomiting.

Part - II

Aim: preparation of caprolactum from cyclohexanone

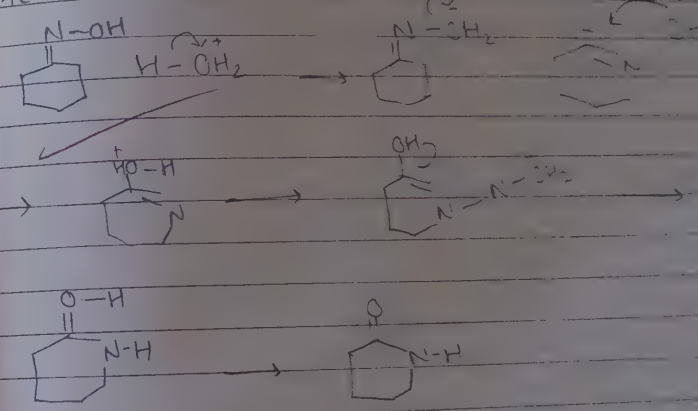
Requirement: Cold water, ice bath, beaker, ... oxime, NaOH etc

Reaction



cyclohexanone oxime → caprolactum.

Mechanism:-



caprolactum.

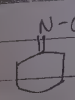
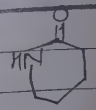
Practical - 2 Preparation

Ex 1 : Preparation of benzil from benzoin 26/04/24

[illegible] conc HNO_3 in conical [illegible]

Stoichiometric	cyclohexanone.	conc H_2SO_4
Molecular formula	$C_6H_{11}NO$	H_2SO_4
Molecular Weight	113	98
Molar ratio	1	1
Φ (gm)	1gm	0.8624
Φ (cm^3)	-	0.468
no of moles	0.0088	0.0088

Spectral Data

	cyclohexanone oxime	caprolactam
Structure		
IR (cm^{-1})	C-H (sp^2) streaching at 3100 - 3000 cm^{-1} C-H (sp^3) streaching at 3000 - 2900 cm^{-1} O-H streaching at 3600 - 3200 cm^{-1}	C-H (sp^2) streaching at 3100 - 3000 C-H sp^3 Streaching 3000-2900 C=O Streaching 1800 - 1660.
^{1}HNMR	Ar-H at region 6.5-8.5 ppm	Ar-H at region 6.5-8.5 ppm.

procedure:-

Take 1g of cyclohexanone oximes add cold solution of conc. H_2SO_4. Warm the reaction mixture gently to bring the reaction to begin the reaction.

Remove the flask from burner and allow the continue. ones the reaction is subsite cool the containt of flask and then 4 cm^3 of water.

mean while prepare a solution of 3.2 g of NaOH in 7.2 cm^3 of water. add prepared solution to the reaction mixture dropwise transfer this cold alkaline solution to the speracting funnel and extract the Compound by Using three portion of 5 cm^3 of carbon tetra Chloride yellow oil obtained which is solidified cooling the purified Compound.

Note the yield of the pure caprolactum. take mp.

Check the purity of by TLC submit the crude product.

Observation:-

Weight of the purified product: 0.8gm.

Melting point :- 66°C

Calculation

Theoritical yield

113gof cyclohexanone oxime = 113 gm of caprolactum

1g of cyclohexanon oxime = x gm of caprolactum

$$x = \frac{113 \times 1}{113}$$

$$x = 1 \text{ gms.}$$

Practical: 1 Preparation of benzoin

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[illegible] in conical [illegible]

Percentage yield: $\frac{\text{Weight of the product}}{\text{Theoritical yield}} \times 100$

$= \frac{0.8}{1} \times 100$

$= 80\%$

Rf value: $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$= \frac{4.5}{3.2} = 1.4$

Result:

Theoretical yield : 1 gms
Weigh of product : 0.8 gms
Percentage yield : 80%
Melting point : 66°C
Rf value : 1.4
Solvent System : ethyl acetate 20%
n-hexane : 80%

MSDS Data:

Compound	Health Hazards	First aid
Caprolactum.	Skin contact Inhalation Ingestion	Wash immediately with soap and remove to fresh clean mouth with water
Carbon tetra Chloride.	eye contact Inhalation Ingestion	Wash immediately plenty of water move to fresh Do not induce vomine
H_2SO_4	eye contact Skin contact Inhalation.	Rinse immediately with plenty of water wash immediately and move to fresh air

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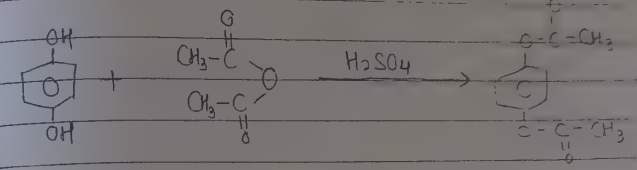
Practical NO 7

Hydroquinone → Hydroquinone diacetate → 2,5 dihydroxyacetophenone

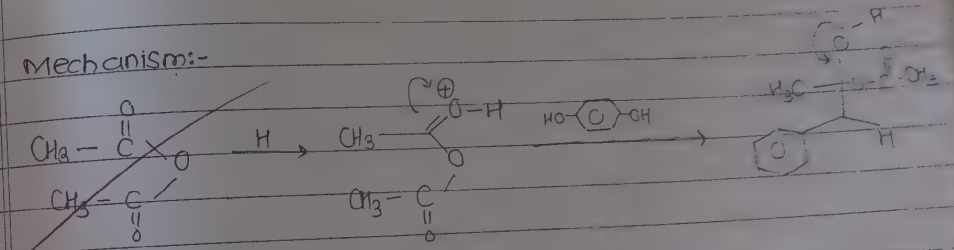
Aim:- preparation of hydroquinone diacetate from hydroquinone.

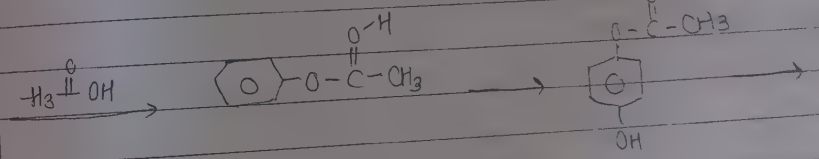
Requirements:- Conical flask, glass rod, Conc H_2SO_4 hydrous zinc acetic anhyside, cold waters.

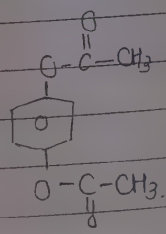
Reaction:-



Mechanism:-







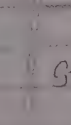
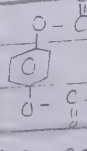
benzoin 26/04/24

conical

Stoichiometric Calculation:-

	Hydroquinone	Acetic anhydride
Molecular formula	$C_6H_6O_2$	$C_4H_6O_3$
Molecular weight	110	102
molar ratio	1	1
Φ (gm)	5	4.59
Volume	–	4.25
no of moles	0.045	0.04

Spectral Data:-

	Hydroquinone	Hydroquinone diacetate.
Structure		
IR (cm^{-1})	3100-3000 cm^{-1} C-H sp^2 Streching 3000-2900 cm^{-1} Streching C-H (sp^3) Streaching 3600-3200 cm^{-1} O-H group	3100-3000 cm^{-1} Sp^2 Streaching 3000-2900 cm^{-1} Streaching 1800-1660 C=O Streaching
^1H NMR	Ar-H 6.5-8.5 ppm R-OH 3-6.5 ppm	Ar-H 6.5-8.5 ppm.

Procedure:-

Add a 1 drop of conc. H_2SO_4 to a mixture of 5 gm of Hydroquinone and 8.6 cm^3 of aceticahyde in conical flask.
Stir the mixture Until dissolve hydroquinone.
After 5 minutes pour the mixture into crushed ice.
Filter with Suction pump and wash with cold water note the yield of hydroquinone diacetate.
purify the small portion of product and take melting point.

Observation:-

Weight of product:- 3.8 gm
Melting point :- 124°C

Theoretical yield:-

110 gm of Hydroquinone = 194 gm of hydroquinone diacetate.
5 gm of Hydroquinone = x

$$x = \frac{194 \times 5}{110}$$

$$\boxed{x = 8.82}$$

percentage yield:-

$$\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$$

$$\frac{5.8}{8.82} \times 100$$

$$= 70\%$$

Result:

Theoretical yield :- 5.82 gm

Practical yield :- 8.82 gm

Percentage yield :- 70%

Melting point :- 104°C

MSDS Data:-

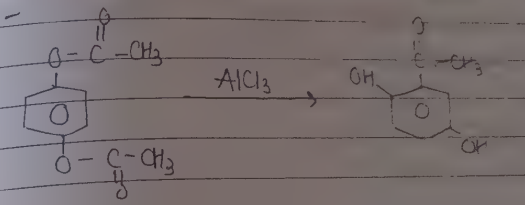
Name of compound	Health Hazard	First Aid.
Hydroquinone	Skin contact Eye contact Ingestion.	Wash immediately with plenty of waters and remove contact lenses immediately flush eyes with waters.
Acetic anhydride	Skin contact eye contact Inhalation	Wash immediately with waters. Take immediately medical treatment flush eyes with plenty of waters.

Part - II

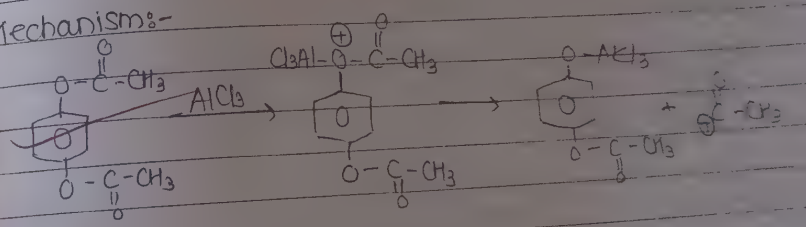
Aim:- preparation of 2,5 dihydroxy acetophenone from hydroquinone diacetate.

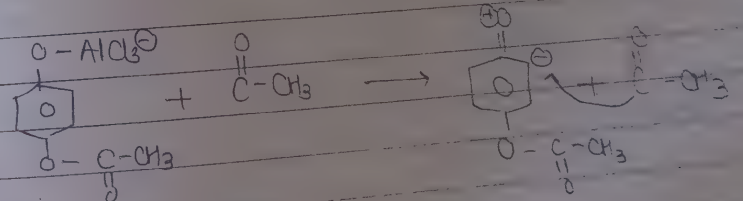
Requirement:- Glass rod, Conical Flask, hydroquinone diacetate 3gm, conc HCl.

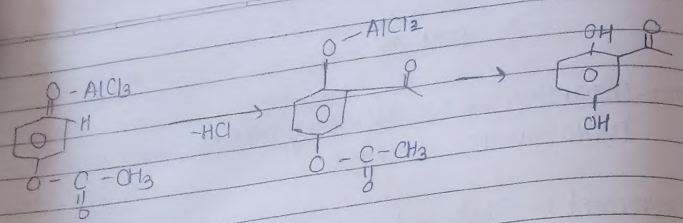
Reaction:-



Mechanism:-



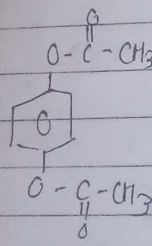
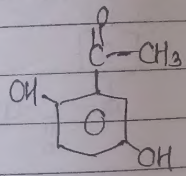




Stoichiometric calculation:-

	Hydroquinone diacetate	$AlCl_3$
Molecular formula	$C_{10}H_{10}O_4$	$AlCl_3$
Molecular weight	194	133.5
Molar ratio	1	1
Q (gm)	1gm	0.629
Volume (cm^3)	-	-
no of moles.	0.0051	0.0051

Spectral Data:-

	Hydroquinone diacetate	2,5 dihydroxy acetophenone.
Structure:		

IR (cm^{-1})	3100-3000 cm^{-1} CH(sp^2) 3000-2900 cm^{-1} CH(sp^3) 1800-1660 cm^{-1} C=O	3100-3000 cm^{-1} CH(sp^2) 3000-2900 cm^{-1} CH(sp^3) 1800-1660 cm^{-1} C=O
^1H NMR	Ar-H 6.5-8.5 ppm	Ar-H 6.5-8.5 ppm.

procedure:-

Take the mixture of 1gm of hydroquinone diacetate and 2.5 gm of anhydrous aluminium chloride in rbf and fitted by air condenser by calculation tube.

Heat the flask in oil bath slowly such that the temperature reaches 100-120 at the end of 30° mint evolution of hydrogen chloride the begins

Now rise the temprature slowly to 160-165°C maintain the tempature for 3 hours.

allow to cool at room temperature.

Add Coushed ice followed by 20° cm HCl in orders delumine to excess of $AlCl_3$.

Filter the product with suction and wash it with cold water

Recrystallise with 99% ethanol

Note the yield of product.

Check the purity by TLC and take melting point

Submit the azied product.

Observation:-
Weight of product:- 0.4
Melting point :- 204°C.

Calculation:-
Theoretical yield:-
194gm of hydroquinone diacetate = 152 gm 2,5 dihydroxy acetophenone
1gm of hydroquinone diacetate = x/152 gm 2,5 dihydroxy acetophenone.

$$x = \frac{194 \times x}{152}$$

$$x = \frac{194}{152}$$

$$x = 1.3 \text{ gms}$$

Percentage yield:-

$$\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.4}{1.3}$$

$$= 30.7\%$$

RF value:- Distance travelled by solute / Distance travelled by solvent

$$= \frac{3.15}{4.2}$$

$$= 0.83$$

Solvent system:- non-polar - 60%
polar - 40%

Result:-

Weight of the products:- 0.4
Theoretical yield :- 1.3gms
Percentage yield :- 30.7%
RF value :- 0.83.
Solvent system :- non-polar - 60%
polar - 40%

Melting point :- 204°C

Observation:-
Weight of product:- 0.4
Melting point :- 204°C.

Calculation:-
Theoretical yield:-
194gm of hydroquinone diacetate = 152 gm 2,5 dihydroxy aceto phenone

1gm of hydroquinone diacetate = x/152 gm 2,5 dihydroxy aceto phenone.

$$x = \frac{194 \times x}{152}$$

$$x = \frac{194}{152}$$

$$x = 1.3 \text{ gms}$$

Percentage yield:-

$$\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.4}{1.3}$$

$$= 30.7\%$$

Rf value:- $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$$= \frac{3.15}{4.2}$$

$$= 0.83$$

Solvent system:- non-polar - 60%
polar - 40%

Result:-

Weight of the products:- 0.4
Theoretical yield :- 1.3gms
Percentage yield :- 30.7%
Rf value :- 0.83.
Solvent system :- non-polar - 60%
polar - 40%

Melting point :- 204°C

MSDS Data:-

Name of Compound	Health Hazard	First Aid.
Hydroquinone diacetate	eyes contanct skin contact	Wash with plenty of water amd move to fresh air.
Aluminium Chloride	eyes contanct skin contact	Wash with plenty of water and take medical treat mentanal move to fresh air

26/9/23